PDU Report NREL/Amoco CRADA Task 1

February 8, 1996

Dates of task: 8/01/95 to 11/1/95
Objective: Prepare the PDU for Operation

1.0 Executive Summary

Task 1 under Phase 3 of the Amoco CRADA, designated to be 3 months long, was created to provide time to finish mechanical shakedown of some of the Process Development Unit (PDU) backend systems and correct design flaws in other systems. The task was carried out in the time period August 1, 1995 to November 1, 1995. As of November 1, 1995, Task 1 was approximately 80% complete. At that time, some of the improvement work was not yet completed due to long lead time items, but the PDU was sufficiently debugged to demonstrate its capabilities, Task 2 in the CRADA Phase 3 plan, To date, Task 1 work is 90% complete with most of the remaining work on hold due to changes in Phase 3 plans.

2.0 Introduction

As of March, 1995, mechanical shakedown of the PDU facility was approximately 50% complete. Initial testing conducted during February and March revealed several areas in the PDU requiring redesign to make the plant operate as intended. Work planned under Phase 3 of the CRADA required use of almost all of the PDU systems, so a period of time was set aside to complete the shakedown and make the necessary improvements. Work lists for each of the main PDU areas were generated by the team and items prioritized. As the work progressed, more items were identified and added to the master work list. Attachment 1 shows a Gannt chart detailing each of the major jobs and their completion to date.

3.0 Discussion

Outlined below is the work identified for each area of the PDU. In most cases, the work was completed prior to the end of Task 1. Mention is made of those jobs that were completed later or are still pending.

3.1 Feedstock Handling/Conveying

Because the Amoco Pretreatment Reactor (APR) was scheduled to be used for the CRADA testing, feedstock handling improvements focused on the APR hopper, and feed screw. Low and high level sensors were added to the APR screw feeder as well as a low level alarm and an access hatch. In addition, a flexible transfer screw was ordered to allow the PDU main feed hopper and conveying system upstream of the Sunds reactor to be used to feed the APR. The flexible conveyor will transfer material from the outlet of the cleated belt conveyor (SC-120) to the APR feed hopper, eliminating manual drum dumping. The transfer screw is scheduled to be in place for Task 4, a continuous run. Some adjustments to the main

PDU feed hopper and interlocks on the feed handling equipment may be necessary before this system is completely integrated with the APR feed system. A dust hood for the main feed hopper was designed, fabricated and installed.

3.2 Pretreatment (APR)

There were several improvements scheduled for the APR based on its performance in 2 runs made in March and April. Major improvements in the condensate system and acid handling system were planned and flow meters to improve the mass balance were ordered. In addition, the metallurgy of some components of the APR were upgraded.

The chiller system was determined to be inadequate for the vapor flow from the APR, so thermocouples were installed to obtain data to better size a replacement system and in the interim the existing system was repiped to a counter-current configuration. A flow meter was planned for this stream, but has not been ordered to date. The need for a larger chiller is still being discussed.

The pre-existing acid make-up and delivery system was labor intensive and difficult to control. J. Farmer designed an acid measuring system to deliver an exact amount of concentrated acid to the mixing tanks, eliminating the need for an iterive batching/titration process. Probes were added to allow real time monitoring of the acid concentration. An elevated acid day tank was added and the line to the APR recessed in the floor to eliminate air entrapment in the acid. Automated fill and low level sensors were installed in the day tank.

Bailey-Fisher-Porter flow meters were ordered for the acid, steam, valve water and flush water lines. When they arrived, they were not as specified and were returned. Micro Motion flow meters were ordered as replacements and were installed. Data from these meters will improve the mass balance closure around the reactor.

In addition to the work on the APR, several improvements in the Sunds pretreatment reactor were conceived and completed, the most notable being better level control in the reactor.

3.3 Fermentation

Several improvements were made in the transfer systems to the fermentation train. Original control valves and flow meters for the enzyme, nutrient and inoculum addition did not control accurately in the low range desired. Several control scenarios were evaluated for each stream. Ultimately, all the original Micro Motion flow meters were either respaned or changed out for the correct flow range including the flow meter controlling the bypass of process fluid around the last fermenter, V455D. A sanitary Baumann valve was chosen for the inoculum control and a Micropump positive displacement pump was specified for the enzyme, due to particulate matter in the enzyme. The nutrient currently used in the PDU, Corn Steep Liquor (CSL), also had particulates in it, so a V-notch ball valve from

Fisher was installed. This valve appeared to be too large for the flow rate, so a pump will be tested during the next run. During Task 2, the inoculum control system and enzyme pump worked well. Some problems were experienced in getting a true measurement from the enzyme and CSL flow meters, possibly due to air entrapment in the transfer lines. Work is ongoing to correct this.

Exhaust gas flow meters on all 6 fermenters (9000L and 1450L) that were equipped with them were respanned. The original flow rate specification for these meters was much lower than current operational rates. In addition, the air control systems on all the seed fermentors (1450L and 160L) were replaced with a NEMA 4 flow element and Baumann control valve. Back pressure valves on all fermenters were adjusted to get them to close more tightly; however, since this valve type (waffle, class 4) is not designed to completely stop gas flow, they may need replacement with a class 6 (globe) type valve.

Condensers for the exhaust gas were specified and ordered to minimize ethanol and other condensible carryover to the scrubber system and a second set of gas flow meters (Kurz) were ordered to allow measurement of exhaust gases during aerobic fermentations. These new components will be installed on the fermenters when the elements arrive and the testing schedule allows.

Load cells were originally installed on the larger fermenters (9000L and 1450L) for level control. The 1450L cells worked well, but the 9000L cells did not give accurate readings due to incorrect initial set-up, rigid piping on all of the external lines, and the pump being mounted to both the tank and the floor. These problems were addressed on all 4 vessels with excellent results. Additional freeing up of piping from the tanks is planned to improve the accuracy of the cells, which will improve the level control (and therefore residence time) in the production train.

Broth recycle was identified early in Phase 3 of the CRADA work and several options discussed to provide a system for the PDU. Among these were rental of a system or use of a NREL lab system currently on order. Because several other decisions had to be made that affected the broth recycle issue, work on it was tabled.

The mass spec became operational during Task 1, reading off-gas composition from V455A only. An upgrade is planned to allow monitoring of all 6 fermenters outfitted with sample gas lines.

In addition to improving the mass balance closure with the changes made to the transfer systems, a flow meter and scale were ordered for the caustic system to allow indirect measurement of the caustic being added to the fermenters. A flow meter installed on the line to the neutralization tank will measure caustic used outside of pH control for the fermenters and the scale will measure total caustic usage.

Since Phase 3 of the CRADA work involved planned use of recombinant organisms in the PDU, part of Task 1 focused on obtaining a Biosafety Level 1 - Large Scale (BL1-LS) rating for the PDU operations and facility. The PDU facility was already designed to achieve BL2-LS rating, so work focused primarily on containment methods and the validation of a kill system to decontaminate the process fluid prior to discharge to the sewer. Since a dedicated kill system was still in the design stage, an interim system using V455D (the last 9000L fermenter) was designed and validated during Task 2. All possible scenarios for spills or decontamination of process fluid from any part of the fermentation train was taken into consideration and several ports were added to allow transfer of fluid into and out of

455D easily and in a contained manner. BL1-LS approval was obtained in January, **1996.**

3.4 Distillation

Work in the distillation system included finishing the mechanical shakedown of the column and support equipment and obtaining approval (in the form of an Operational Readiness Review) from the NREL Environmental Safely and Health group to operate it. Of primary concern was that the fire suppression system be operational and verified. In addition, the entire distillation building was classified as a Division 1, class 1 area and operators were trained in appropriate measures to take when working under that classification.

Once approval to operate was obtained, M. Ruth worked with the DACS system administrator to test and tune the controls and safety interlocks placed on the control system. Starting with water only and proceeding to dilute ethanol and finally hydrolyzate, the distillation system was systematically checked out and improved at each step. Several flushing ports were added at key points of the piping on the skid to allow for cleaning plugs that occurred when distilling the hydrolyzate. The more fermented (digested) the process stream from fermentation, the less problems were encountered. The majority of the plugs were in valves and heat exchanger tubing to and from the column. When poorly hydrolyzed sawdust was put into the system, several plugs brought the system down. Additional water supply lines are proposed for the skid to lessen downtime due to plugging.

A Clean-In-Place (CIP) system was added to the APV skid system to clean the preheaters of gypsum deposits anticipated when running hydrolyzate neutralized with lime. The system was tested, but Phase 3 plans do not currently include the use of lime.

The ethanol storage tank flow meter was tested and the ethanol loadout system scheduled for a second test, still to be completed.

3.5 Centrifugation/Water Recycle

The least operated sections of the PDU, Centrifugation and Water recycle, passed mechanical shakedown but were not improved beyond the original design. Additional work on the flow control element on the feed line to the centrifuge was to determine its operability through a range of hydrolyzate homogeneity. The valve is sufficient for well digested material, but problems occurred with undigested or partially digested material plugging the control valve. The centrifuge was tested several times and provided a range of solids percentage (2040%) depending on the feed to the system, the backdrive speed and dam height. When representative material is produced (likely in Task 2 and 3), these parameters will be fine-tuned. Maintenance on the centrifuge is extensive and needs to be taken into account during the planning of the Task 3 run.

The water recycle system, designed to sterilize the effluent stream from centrifugation and return it to the process via flash tank water addition, has yet to be completely checked out. It was considered lowest priority of the Task 1 work and will be picked up during or between Tasks 2, 3 and 4. Work includes testing the control of the system and validating its sterility.

3.6 Utilities

Extensive work was planned to upgrade the PDU utilities independent of the Amoco CRADA and the timing of several of these projects coincided with Task 1. The most major upgrade was to **the** Cooling Water **(CW)** system and necessitated erection of 2 mezzanines to hold the equipment. The new CW system, designed to deliver **4** times the capacity and 3 times the heat removal, should provide adequate cooling capacity for any plant additions in the future. A new 6" CW header system was installed and where possible, the old system tie-ins were utilized to cut time and cost. A new air compressor was also installed during Task 1 to provide **the** PDU with its own air supply. The Atlas-Copco compressor provides enough air to supply both the plant (30 psig) and instrument (80 psig) air needs. A separate Champion compressor provides air to the APR with a backup Champion unit provided temporarily free of charge onsite due to problems experienced with the original unit.

During Task 1 tie-ins to the DACS of several crucial utility monitoring points were completed. Others were initiated and are awaiting components or DACS system administrator time to be completed. Monitoring of both the plant and instrument air header pressures, steam pressures in both the high and low headers and general failure signals from **the** boiler and new CW system are scheduled to be completed. Several signals from the scrubber system were terminated at the **DACS** and a screen added to allow the PDU operator to monitor pH, Oxidation Reduction Potential (ORP), fan and circulation pump discharge pressure, and sump level alarms.

A CIP system was designed and ordered to reduce manpower associated with cleaning the PDU numerous **PDU** vessels. Expected delivery is February, 1996. A mezzanine extension is planned on the east side of the PDU to place the CIP system.

A chemical storage shed was located on the north side of the building, under the CW tower mezzanine. This shed has made the PDU more efficient in handling its chemical inventory, since it no longer needs to use the ES&H group's storage facility. The shed has a **HVAC** unit for climate control and separate containment dikes for acid and base.

Chilled Water (CHW) load for the vent condensers planned for the fermenters was specified by Stone & Webster engineers. A unit already onsite is planned for refurbishment to provide this utility. **A** hot water system was ordered and installed to provide hot (150 °F) water to the plant floor for washing, batching, etc. This unit should reduce energy usage as it is **a** more efficient way to heat water than in **a** jacketed vessel.

Back-up power for the PDU will be provided by a generator already at NREL. Installation and testing of the unit **is** slated to be complete by January, 1996. An Uniterruptable Power Supply **(UPS)** for the DACS is also scheduled to be installed and tested by that time.

3.7 Data Acquisition and Control System (DACS)

DACS work included outstanding support of all the equipment improvements occurring in the PDU to tie them into the control system as well as ongoing work to improve the control scheme itself. ***An*** Alarm Queue was initiated to provide better notification to the operators of events that required action such as a low level in a batching tank. Several corrections to the instrumentation eliminated the need for constant supervision of equipment. Others improved the data coming from the instrumentation.

4.0 Conclusion

The primary goal of Task 1, get the PDU operational, has been achieved. There is always room for improvement, but the mechanical component of the process is working, as evidenced by the recent completion of a 30 day continuous run utilizing all but the water recycle system. While all the tasks identified in Task 1 are not yet complete, all are in progress. The limiting factor is resources, be it manpower or money. Because the PDU is a constantly evolving and changing process subject to the demands of its users, it is conceivable that some of these improvements may become higher or lower priority and as such may see quick completion or take a back seat in favor of another job. Monitoring and prioritizing of these tasks as well as new ones identified as the PDU operates is an ongoing process.

5.0 Acknowledgments

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